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OPTIMIZATION OF THE PRODUCTION OF DIETARY FIBER CONCENTRATES FROM BY-PRODUCTS OF PAPAYA (*CARICA PAPAYA* L. VAR. FORMOSA) WITH MICROWAVE ASSISTANCE. EVALUATION OF ITS PHYSICOCHEMICAL AND FUNCTIONAL CHARACTERISTICS

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ABSTRACT

A process involving an ethanol extraction step and microwave assisted dehydration was designed to produce dietary fiber concentrates (DFC) from papaya (*Carica papaya* L. var. Formosa) by-products. It was concluded that a 15 min extraction with 2.9 mL of ethanol/g of papaya pulp followed by drying performed at 40C, produced DFC with optimal values for functional properties (hydration properties, oil holding capacity, specific volume, water soluble fraction) as well as b^* color parameter and content of phenolic compounds. The DFC obtained from papaya peel using the same conditions, showed a higher content of cell wall polysaccharides and higher glass transition temperature (38C) than the one obtained from pulp (8C) and the polyphenol content doubled the one of pulp. Both DFCs showed potential to be used for nutritional purposes as well as for technological applications as antioxidants and/or thickeners.

PRACTICAL APPLICATIONS

Consumer demands for more healthy food products and global policies on the issues of health and environment are the keys for the development of innovative food products with profitable markets. The conversion of by-products from the papaya industrialization to dietary fiber concentrates (DFC) with functional activity fulfills these requirements, being salad dressings or dairy products, the targets for DFC application as viscosity modifiers and nutritional value improvers.

INTRODUCTION

By-products are generated in the different stages of food processing. These products consist of peels, seeds and parts of the pulp in the case of fruit and vegetables. Moreover, plant tissues that do not meet the quality requirements of the industry and the market, are disposed as wastes from agricultural feedstocks. The transformation of plant wastes into value added products can help to retrieve valuable biomass providing the possibility of a better use of this important source of nutrients (Laufenberg *et al.* 2003). Es. . .., new version. These wastes can be used to produce valuable compounds such as lactic acid obtained through fermentation processes (Pagana *et al.* 2014; Panesar and Kaur 2015), phenolic compounds with antioxidant activity and polyunsaturated fatty acids (Bordiga *et al.* 2015; Iora *et al.* 2015). The production of dietary fiber (DF) from these residues is another way of adding value while giving origin to a nutritional ingredient to be incorporated in food (de Escalada Pla *et al.* 2007; de Escalada Pla *et al.* 2010; de Escalada Pla *et al.* 2012).

Several studies have shown that a diet with an adequate intake of DF is associated with a low incidence of some chronic diseases such as obesity, diabetes mellitus, colon cancer, cardiovascular disease, colonic diverticulitis and constipation (Eshak *et al.* 2010; Isken *et al.* 2010). Insoluble fiber can absorb, swell and entrap water within its porous matrix and water retention properties contribute toward the bulking effect of fiber in the colon. They can take part in the dilution of cytotoxic substances in the large intestine, thus reducing harmful potency (Guillon *et al.* 2011).

Fractions enriched in DF can be incorporated into food products as low caloric ingredients, for the partial replacement of flour, sugar or fat, as enhancers for the retention of water or oil, to improve the stability of emulsions or to preclude oxidation processes (Elleuch *et al.* 2011). Functional properties of DF are related not only to the source but also to the process conditions implicated in its extraction (Guillon *et al.* 2011). For instance, de Escalada Pla *et al.* (2010) observed that functional properties of fiber obtained from quince industrialization wastes, varied with drying conditions. Nieto Calvache *et al.* (2015) demonstrated that when an ethanol pre-treatment and a microwave drying were used for fiber obtention from peach bagasse, the properties could be modulated with the change in the conditions used for both steps applied.

Carica papaya is considered the most important edible fruit of the *Caricaceae* family (Wurochekke *et al.* 2013). The plant grows in tropical and sub-tropical regions and its fruit is rich in antioxidants such as polyphenols, vitamins and carotenoids (Rivera-Pastrana *et al.* 2010). Asia is the main producer of papaya in the world followed by South America, Africa and Central America. The market demand for tropical fruits has been growing steadily over the past years, and papaya has gained worldwide popularity. The U.S.A. is currently the largest papaya importer because of its high per-capita income (Evans and Ballen 2012). It is cultivated mainly for the fruit use as such for breakfast or for the production of jellies, preserves and juices. It is also the source of papain, the proteolytic enzyme with many industrial uses (Oloyede 2005).

The aim of this study was to evaluate a technique proposed for the production of DFC from papaya residues. For this goal, it was evaluated the significance of different variables [extraction time *t*, extraction temperature *T*, ethanol/sample ratio *E/S* and drying temperature *Td*] related to the steps of extraction and drying involved in the process. It was also studied the levels of the significant variables that allow to improve the functional properties (hydration properties, oil holding capacity [OHC], water-soluble fraction [WSF], specific volume), color parameters (L^*, a^*, b^*) and phenolic compounds of the DFC obtained from pulp. The characteristics of peel DFC obtained in the same conditions were also evaluated and the determination of yield, alcohol insoluble residue (AIR) content and glass transition temperature of DFCs deepened the characterization of their technological potential.

MATERIALS AND METHODS

DFC Preparation

Papaya fruits were purchased in a local market of Buenos Aires city, Argentina.

In a first step, the pulp and peel were separated. Then, the extraction of cell wall polysaccharides was performed by subjecting the pulp to different ethanolic treatments according to the experimental design (Table 1). A mechanical homogenizer at 10,000 rpm (Omni Mixer) was used for the extractive treatment. Subsequently, the mixtures were filtered and the solid residue was placed in polypropylene trays (15 × 10 × 5) cm, with a bed height of 1 cm, for the following drying step. Dehydration of DFCs was carried out with an Ethos Plus microwave equipment (Milestone, Italy) working at a maximum power of 450 W and at different temperatures according to the experimental design (Table 1). The drying was conducted until constant weight was achieved. Additionally, water activity (A_w) was measured to corroborate that it reached values below 0.6 to assure product stability (Muggeridge and Clay 2001). T1

The dried DFC was milled and sieved through a mesh ASTM 40 to obtain particles of sizes below 420 microns. Samples of each DFC, were vacuum packed in Cryovac bags (Sealed Air Corporation, Argentina) and stored at −18C until their characterization.

Evaluation of the Functionality of DFC

All determinations of the properties described below were performed in triplicate.

Hydration properties such as: water-holding capacity, WHC; swelling capacity, SC; water retention capacity, WRC; retained water, RW as well as OHC were determined as previously described by de Escalada Pla *et al.* (2010).

The water soluble fraction, WSF of DFC was determined on the supernatant of the WRC assay after its freeze drying. The WSF was calculated as:

$$\mathrm{WSF}(g/100g)=\frac{M1}{M2}*100$$

where M_1 is the mass of solids in the freeze dried supernatant and M_2 is the mass initially weighed of DF fraction on dry basis.

Physicochemical Characterization of the DFC

Apparent Density and Specific Volume. Apparent density was determined by measuring the volume of a weighed sample (≈2 g) in a 5 mL graduated and calibrated tube. The specific volume was determined as the inverse function of the apparent density.

TABLE 1. COMPLETE FACTORIAL DESIGN (2^4) AND CENTRAL POINTS. CODED AND UNCODED LEVELS FOR THE FOUR FACTORS ASSAYED AND RESPONSES RECORDED FOR THE TWELVE VARIABLES MEASURED ON DFC FROM PAPAYA

Extraction time (min)	Extraction temperature (C)	Ethanol/ sample ratio (mL/g)	Drying temperature (C)	WHC (g/g)	SC (mL/g)	WRC (g/g)	RW (g/100g)	OHC (g/g)	Specific volume (mL/g)	WSF (g/100g)	Yield (g/100g)	*L**	*a**	*b**	Total polyphenols (g/100g)
45 (1)	80 (1)	5 (1)	30 (−1)	31 ± 1	25.8 ± 0.2	21.3 ± 0.5	49.6 ± 0.1	0.90 ± 0.03	1.44 ± 0.01	20.0 ± 0.2	3.704 ± 0.009	54.35 ± 0.04	23.69 ± 0.03	43.92 ± 0.05	0.324 ± 0.006
45 (1)	80 (1)	2 (−1)	70 (1)	20 ± 1	16.1 ± 0.8	14.74 ± 0.02	34.5 ± 0.3	0.92 ± 0.02	1.428 ± 0.009	21.1 ± 0.7	3.11 ± 0.01	54.5 ± 0.1	30.93 ± 0.09	45.8 ± 0.2	0.2944 ± 0.0002
45 (1)	20 (−1)	5 (1)	70 (1)	31 ± 2	26.2 ± 0.7	18.9 ± 0.6	47.1 ± 0.4	1.64 ± 0.03	2.141 ± 0.009	16 ± 1	2.90 ± 0.01	64.26 ± 0.05	23.39 ± 0.01	27.26 ± 0.03	0.35 ± 0.01
45 (1)	20 (−1)	2 (−1)	30 (−1)	38.2 ± 0.9	37.4 ± 0.7	24.5 ± 0.7	67.4 0.9	1.02 ± 0.03	1.51 ± 0.02	8.9 ± 0.9	2.70 ± 0.01	53.79 ± 0.02	36.46 ± 0.02	45.74 ± 0.04	0.378 ± 0.009
15 (−1)	80 (1)	5 (1)	70 (1)	34.7 ± 0.7	31.7 ± 0.7	21.0 ± 0.7	55.6 ± 0.9	1.26 ± 0.04	1.61 ± 0.05	13.89 ± 0.04	2.76 ± 0.01	72.59 ± 0.01	16.80 ± 0.02	24.773 ± 0.006	0.35 ± 0.01
15 (−1)	80 (1)	2 (−1)	30 (−1)	37.1 ± 0.6	35.3 ± 0.3	21.7 ± 0.6	59.4 ± 0.7	1.09 ± 0.03	1.59 ± 0.02	11.47 ± 0.07	2.83 ± 0.01	57.48 ± 0.01	31.60 ± 0.02	48.10 ± 0.05	0.27 ± 0.02
15 (−1)	20 (−1)	5 (1)	30 (−1)	79.2 ± 0.4	90.8 ± 0.2	28.5 ± 0.2	80 ± 2	1.326 ± 0.002	1.839 ± 0.003	3.7 ± 0.6	3.14 ± 0.01	64.24 ± 0.03	27.03 ± 0.03	29.57 ± 0.04	0.297 ± 0.007
15 (−1)	20 (−1)	2 (−1)	70 (1)	20.5 ± 0.5	15.5 ± 0.1	14.5 ± 0.7	35.7 ± 0.3	0.98 ± 0.03	1.51 ± 0.01	21.0 ± 0.9	2.88 ± 0.01	56.85 ± 0.05	31.55 ± 0.01	45.09 ± 0.08	0.368 ± 0.007
45 (1)	80 (1)	5 (1)	70 (1)	44.8 ± 0.4	43.5 ± 0.1	22.8 ± 0.2	56.3 ± 0.1	1.21 ± 0.05	2.12 ± 0.01	15.4 ± 0.7	2.75 ± 0.01	78.71 ± 0.03	6.755 ± 0.007	25.15 ± 0.03	0.314 ± 0.006
45 (1)	80 (1)	2 (−1)	30 (−1)	39 ± 1	34.8 ± 0.8	21.9 ± 0.6	56.8 ± 0.4	0.9408 ± 0.0001	1.71 ± 0.02	9.9 ± 0.6	2.79 ± 0.01	59.4 ± 0.1	23.32 ± 0.01	45.9 ± 0.2	0.27 ± 0.01
45 (1)	20 (−1)	5 (1)	30 (−1)	54.0 ± 0.9	54.4 ± 0.8	20.7 ± 0.9	50.8 ± 0.3	1.13 ± 0.06	2.008 ± 0.007	10.0 ± 0.6	2.75 ± 0.01	64.75 ± 0.06	21.83 ± 0.03	28.535 ± 0.007	0.292 ± 0.03
45 (1)	20 (−1)	2 (−1)	70 (1)	38.4 ± 0.2	32.5 ± 0.2	20.3 ± 0.4	57 ± 1	0.93 ± 0.02	1.74 ± 0.04	8.4 ± 0.2	2.63 ± 0.01	57.375 ± 0.007	30.37 ± 0.01	48.77 ± 0.04	0.308 ± 0.006
15 (−1)	80 (1)	5 (1)	30 (−1)	74.0 ± 0.8	89.1 ± 0.6	23 ± 1	65.6 ± 0.9	1.47 ± 0.05	2.46 ± 0.05	6.6 ± 0.5	2.66 ± 0.01	82.74 ± 0.00	3.62 ± 0.02	19.43 0.02	0.3087 ± 0.0005
15 (−1)	80 (1)	2 (−1)	70 (1)	31.8 ± 0.3	25.3 ± 0.2	18.8 ± 0.6	48 ± 1	1.01 ± 0.05	1.70 ± 0.03	11.7 ± 0.4	2.65 ± 0.01	61.6 ± 0.1	23.61 ± 0.04	45.5 ± 0.1	0.317 ± 0.002
15 (−1)	20 (−1)	5 (1)	70 (1)	44.03 ± 0.05	40.8 ± 0.1	27 ± 2	74.6 ± 0.9	1.11 ± 0.03	1.90 ± 0.03	9.9 ± 0.1	2.83 ± 0.01	63.14 ± 0.01	25.38 ± 0.00	38.56 ± 0.06	0.348 ± 0.006
15 (−1)	20 (−1)	2 (−1)	30 (−1)	46.1 ± 0.5	43 ± 1	24.1 ± 0.8	66.7 ± 0.9	0.83 ± 0.04	1.56 ± 0.04	8.1 ± 0.9	3.02 ± 0.01	54.88 ± 0.03	30.02 ± 0.02	45.11 ± 0.03	0.359 ± 0.004
30 (0)	50 (0)	3.5 (0)	50 (0)	34.8 ± 0.8	31.23 ± 0.09	20.7 ± 0.8	53.7 ± 0.8	1.32 ± 0.03	1.81 ± 0.02	15.0 ± 0.4	2.82 ± 0.01	60.06 ± 0.04	29.35 ± 0.03	24.25 ± 0.03	0.31 ± 0.02
30 (0)	50 (0)	3.5 (0)	50 (0)	45 ± 1	37.1 ± 0.6	24.6 ± 0.5	68.9 ± 0.8	1.24 ± 0.05	1.77 ± 0.02	9.0 ± 0.9	2.89 ± 0.01	61.17 ± 0.03	27.99 ± 0.05	26.97 ± 0.02	0.309 ± 0.006
30 (0)	50 (0)	3.5 (0)	50 (0)	45.0 ± 0.3	38.5 ± 0.8	25.2 ± 0.5	70.0 ± 0.9	1.53 ± 0.04	2.004 ± 0.004	7.7 ± 0.2	2.84 ± 0.01	64.77 ± 0.01	25.62 ± 0.03	22.71 ± 0.06	0.410 ± 0.008
30 (0)	50 (0)	3.5 (0)	50 (0)	45.0 ± 0.3	40.0 ± 0.2	25.6 ± 0.5	71 ± 2	1.34 ± 0.01	1.874 ± 0.009	9.7 ± 0.8	2.89 ± 0.01	63.19 ± 0.01	27.036 ± 0.006	26.43 ± 0.01	0.367 ± 0.007

Responses are informed as the mean and standard deviation ($n = 3$).

WRC: Water retention capacity, RW: Percentage of water retained, WHC: Water-holding capacity, SC: Swelling capacity, OHC: Oil holding capacity, WSF: Water soluble fraction.

Moisture Content and Water Activity. Moisture was determined on ≈ 0.500 g sample using infrared heating (Ohaus MB45 moisture analyzer Corporation) till constant weight. The A_w was determined using an AQUA LAB Series 3 Quick hygrometer (Start Decagon Devices, Inc.). AQ4

Alcohol Insoluble Residue. AIR was obtained by consecutive treatments with boiling ethanol (96 mL/100 mL) of the DFC obtained with the optimized process and galacturonic acid content was determined on the AIR obtained (de Escalada Pla *et al.* 2007).

Differential Scanning Calorimetry. The glass transition temperature, T_g, was determined by differential scanning calorimetry, DSC, by means of a Mettler Toledo 822 equipment and STARe Thermal Analysis System version 3.1 software (Mettler Toledo AG, Switzerland). The instrument was calibrated using standard compounds (indium and zinc) of defined melting point and heat of melting. The measurements were performed in duplicate with 14–17 mg of each sample, using hermetically sealed aluminum pans of 0.04 mL inner volume (Mettler) which were heated from −80 to 80C at 10C/min rate; an empty pan was used as a reference. The confidence interval estimated for temperature was 2C.

Determination of Phenolic Compounds. Determination of total phenolics was carried out according to Bunzel *et al.* (2000). Briefly, 0.9000 g of DFC were mixed, under vacuum, with 1 mol/L NaOH solution for 18 h at 25C. Then, pH was adjusted with HCl to approximately 2. After centrifugation, total phenolics were determined on supernatant by Folin Ciocalteau using gallic acid as standard (Shui and Leong 2006).

Color Evaluation. The color of DFCs was measured in triplicate using a photocolorimeter (Minolta, Japan) and the $L^*a^*b^*$ space defined by the Commission Internationale de l'Eclairage, CIE. The co-ordinates of this space are L^*, the lightness; a^*, the grade of greenness/redness and b^*, the grade of blueness/yellowness. Each sample was placed onto a white tile and values of CIE color space co-ordinates were acquired using illuminant D65 and 2° observer angle.

Determination of Yield. The DFC yield (g/100g) was determined as the ratio between mass of concentrate obtained after the microwave drying and mass of papaya pulp used.

Experimental Design and Statistical Analysis. In the first part of this study, the effect of four factors: time *t* and temperature of extraction *T*, ethanol/sample ratio *E/S* and drying temperature *Td* on DFC properties was studied according to a complete factorial design (2^4) at two levels for each factor and considering central points (Montgomery 2008) which were performed in quadruplicate. Response variables were: hydration properties (WRC, WHC, RW, SC), OHC, specific volume, WSF, color parameters (L^*, a^*, b^*) and content of phenolic compounds. The experimental design, with coded and uncoded values, is shown in Table 1 and was performed in order to identify the factors presenting major effects on the properties studied.

In a second stage of this study, a response surface design (Box Behnken model) was proposed, in order to evaluate the effect of three factors: extraction time *t*, ethanol/sample ratio *E/S* and drying temperature *Td* on DFC properties (Montgomery 2008). The experimental data were fitted to a second degree polynomial function:

$$Y = b_o + \sum_{i=1}^{k} b_i X_i + \sum_{i=1}^{k} b_{ii} X_i^2 + \sum_{i<j}^{k} b_{ij} X_i X_j + e$$

Where, Y is the response variable, b_0 is the intercept value, b_i ($i = 1, 2...k$) is the first-order model coefficient, b_{ij} is the interaction effect, and b_{ii} represents the quadratic coefficients of X_i. X_i and X_j are the input variables (factors) that influence the response variable, and *e* represents the random error (Betiku and Taiwo 2015). Finally, using a multiple response analysis, optimal process conditions were defined. The criteria used for this procedure were to obtain the highest possible values for the analyzed properties. This analysis was performed by means of the desirability function which is one of the most used techniques to optimize multiple responses (de Barros *et al.* 2003). The basic idea of this function is to transform a multi-response problem into a problem with a unique response through mathematic transformations (Del castillo *et al.* 1996). An analysis of variance (ANOVA) was conducted to verify which factors affect significantly the properties analyzed and to check the proportion of variance explained by the proposed model through the determination of the R^2 coefficient. The adequacy of the model was also evaluated through the lack of fit test ($P > 0.05$).

The software Statgraphics Centurion XV (02/15/06 V, 2007) was used in the experimental design and statistical treatment of data.

RESULTS AND DISCUSSION

Complete Factorial Design

The results for all properties examined within the first experimental design are presented in Table 1. The values obtained ranged from 20 to 79.2 g/g for WHC, from 15.5 to 90.8 mL/g for SC, from 14.5 to 28.5 g/g for WRC and from

34.5 to 80 g/100g for RW. For OHC, specific volume, WSF and yield, the values ranged between 0.83 and 1.64 g/g, 1.428 and 2.46 mL/g, 3.7 and 21.1 g/100g and 2.63 and 3.704 g/100g, respectively. Conversely, for the color parameters L^*, a^* and b^*, the values obtained ranged between 53.79 and 82.74, 3.62 and 36.46 and 19.43 and 48.77, respectively. Finally, the content of phenolic compounds took values between 0.27 and 0.41 g/100g.

The significance of regression coefficients, R^2 coefficient and the test of lack of fit obtained by ANOVA are shown in Table 2. It can be observed that for all properties studied, the values of R^2 were higher than 72% and, moreover, P values (lack of fit test) were higher than 0.05, showing that the model proposed explained a percentage higher than 72% of the variability of response properties and that the model was adequate (confidence level: 95%).

The factors: t and E/S as well as their interaction, significantly affected all hydration properties, while Td had a significant effect ($P < 0.01$) on WHC and SC and T had a significant effect ($P < 0.01$) only on SC. The effects of interactions between various factors for SC were also observed with a confidence level greater than 95%.

Conversely, E/S had also a significant effect ($P < 0.05$) on the properties OHC and specific volume, while Td had a significant effect on WSF. Some interaction effects also affected WSF significantly ($P < 0.05$).

At least two factors had a significant effect on the color parameters (L^*, a^*, b^*). A strong effect ($P < 0.01$) of E/S was observed on the three parameters and Td ($P < 0.05$) affected a^*. In addition, there are effects of the interaction between factors on these properties with confidence levels of, at least 95%. Finally, the phenolic compounds were significantly affected by the extraction temperature T and E/S ratio with a confidence level of 95%, and by several interactions ($P < 0.05$).

In this preliminary analysis, it could be verified that the factors related to extraction step that exerted the greatest influence on the properties studied were E/S and t. Conversely, T and drying temperature Td affected an equal number (4) of properties. With the purpose of evaluating the optimum process conditions that render DFCs with the highest values for each of the properties of interest, a fixed value of 20C (lowest level) was fixed for the extraction temperature. In this way, the extraction temperature was set at the lowest level, considering that when its effect was significant on any response, this effect was negative. Accordingly, subsequent response surface analysis, only included three factors.

Box Behnken Design

The results obtained with the response surface design are shown in Table 3. In this new design, drying temperature took values of 40, 60 and 80C because when the drying was performed at a temperature of 30C (lower level) in the complete factorial design, the time required to reach constant weight, was too long, and therefore not recommended from energetic viewpoint (drying times not shown).

The results obtained with this new design for hydration properties ranged between 33.5 and 89 g/g for WHC, 27.1 and 90.6 mL/g for SC, 31.33 and 39.5 g/g for WRC and 47 and 66.3 g/100g for RW. For OHC, specific volume, WSF and yield the results obtained were between 1.02 and 1.40 g/g, 1.50 and 1.83 mL/g, 13 and 26 g/100g and 2.44 and 2.93 g/100g, respectively. Color parameters L^*, a^* and b^* showed the following ranges 53.665–62.43, 26.66–32.29 and 28.88–44.53, respectively. The content of phenolic compounds ranged from 0.368 to 0.48 g/100g.

The coefficients of equations describing the response surfaces for each property and the variance analysis are shown in Table 4. It can be observed that the R^2 for the hydration properties explained at least 81.10% of the variability for these properties, while for OHC, specific volume, WSF and phenolic compounds they explained a percentage higher than 79.28. As for the color, the parameter b^* had an R^2 of 98.81% while L^* and a^* had lower R^2 values (62.19% and 63.87%, respectively). For all properties, the lack of fit test showed P values higher than 0.05, which means that the proposed statistical models were suitable for the explanation of observed data with a 95% confidence level.

Figure 1 shows response surface plots for hydration properties, using E/S and Td as variables, while the t was set at the lowest level (15 min) considering that it was significant only for total polyphenol content (Table 4), and that a lower time will contribute to the optimization of energy costs.

The WHC and the SC properties presented the same trend. A strong and significant negative effect of the Td on these two properties can be observed in Table 4. Both properties were highly influenced by the quadratic coefficient of Td and by the interaction between E/S and Td. Figure 1 (Panels a, b) shows that when the E/S was low, both properties decreased when the Td increased. At high E/S ratios, the same trend was observed until ≈ 60C; from this point and up to 80C, the trend reversed due to the positive quadratic effect (Table 4) previously mentioned. Probably, tissue collapse and shrinkage triggered by high Td, determined changes in the porosity and rehydration capacity (de Escalada Pla *et al.* 2010; Nieto Calvache *et al.* 2015). Collapse and tissue shrinkage occurrence during drying can also be associated to the presence of low molecular weight compounds such as free sugars (Gerschenson *et al.* 1981; Guillon *et al.* 1998) which could not be efficiently removed from the matrix with low E/S ratios.

As can be observed in Table 4, WRC and RW showed similar trends, being these properties significantly and negatively affected by Td and positively affected by the

TABLE 2. COMPLETE FACTORIAL DESIGN. COEFFICIENTS OF REGRESSION AND ANOVA FOR PROPERTIES OF DFC FROM PAPAYA PULP

	WHC (g/g)	SC (mL/g)	WRC (g/g)	RW (g/100g)	OHC (g/g)	Specific volume (mL/g)	WSF (g/100g)	L*	a*	b*	Total polyphenols (g/100g)
Independent term	42.9463	37.0718	23.4042	63.0998	1.17267	1.82826	9.68089	61.4022	26.7799	32.35	0,422448
A: Extraction time	−5.95849*	−9.40667**	−2.61658*	−9.91597*	0.0130127	−0.0565681	0.742121	−3.01261*	0.375	−3.23542*	0,000699173
B: Extraction temperature	−0.758385	−5.55321*	0.957077	2.36597	−0.0482731	0.0431472	0.725546	1.26718	−1.65346*	−4.78625*	−0,00216013*
C:Ethanol sample ratio	9.13818**	7.05227**	3.19378*	9.15535*	0.10787*	0.22604**	0.451079	4.18155**	−5.51417**	−12.4702**	−0,0340796*
D: Drying temperature	−6.77558*	−14.3045**	−1.57471	−4.87609	0.0598329	0.0536451	1.67905*	−0.279904	−3.00154**	0.964896	0,000214575
AB	−2.37849	−4.32023*	−1.41655	−5.4916	−0.121159*	−0.129907*	−1.08311	−3.14636*	0.616042	−2.23229	8,46093 E − 7
AC	−6.07083*	−9.49341**	−2.9193*	−10.6447*	−0.049781	−0.0596196	4.43167**	−2.3049*	−2.54383*	−3.52583*	0,000121921
AD	3.24027	3.93076*	0.115985	1.19047	0.103018*	0.0412051	−3.33774*	0.363221	−1.11146	−0.508646	−0,0000297861*
BC	1.05864	−3.31979*	1.72926	5.8916	0.00202433	0.0299078	2.95626*	−0.0042789	−1.66813*	−4.8725*	0,000374923*
BD	3.73226	−0.364823	0.578957	1.49328	−0.0598793	0.00615805	−3.12536*	−0.774904	−2.42049*	−0.375313	0,00000546
CD	−0.640967	−6.55138**	1.41268	4.48578	−0.0107518	0.0522238	1.91203*	−0.879696	−0.00145838	0.917396	0,000253868*
R^2	86.48	95.66	87.77	86.09	81.78	72.84	97.09	79.26	92.47	84.47	74,67
Lack of fit (p)	0.1565	0.1565	0.6578	0.3745	0.1010	0.0782	0.4371	0.0619	0.1121	0.0643	0,0596

Significance levels (α): *0.05; **0.01.

WRC: Water retention capacity, RW: Percentage of water retained, WHC: Water-holding capacity, SC: Swelling capacity, OHC: Oil holding capacity, WSF: Water soluble fraction.

TABLE 3. BOX-BEHNKEN DESIGN: CODED AND UNCODED LEVELS FOR THE THREE FACTORS ASSAYED AND RESPONSES RECORDED FOR THE TWELVE VARIABLES MEASURED ON DFC FROM PAPAYA PULP

Extraction time (min)	Ethanol/ sample ratio (mL/g)	Drying temperature (C)	WHC (g/g)	SC (mL/g)	WRC (g/g)	RW (g/100g)	OHC (g/g)	Specific Volume (mL/g)	WSF (g/100g)	Yield (g/100g)	L*	a*	b*	Total polyphenols (g/100g)
15 (−1)	2 (−1)	60 (0)	40.4 ± 0.3	37.21 ± 0.08	35 ± 1	51.8 ± 0.7	1.28 ± 0.03	1.73 ± 0.02	16.3 ± 0.2	2.74 ± 0.04	57.91 ± 0.06	29.23 ± 0.05	44.53 ± 0.05	0.44 ± 0.01
45 (1)	2 (−1)	60 (0)	46.4 ± 0.1	45.7 ± 0.2	36.6 ± 0.1	53.6 ± 0.2	1.20 ± 0.02	1.73 ± 0.01	20 ± 1	2.44 ± 0.04	56 ± 1	31.43 ± 0.03	43.6 ± 0.1	0.459 ± 0.009
15 (−1)	5 (1)	60 (0)	33.5 ± 0.4	27.1 ± 0.4	33.6 ± 0.8	48.3 ± 0.4	1.105 ± 0.002	1.50 ± 0.02	26 ± 2	2.77 ± 0.04	54.26	29.63 ± 0.06	32.19 ± 0.06	0.47 ± 0.03
45 (1)	5 (1)	60 (0)	49.8 ± 0.3	48.2 ± 0.3	38.7 ± 0.8	55 ± 1	1.26 ± 0.02	1.72 ± 0.01	17.9 ± 0.4	2.62 ± 0.04	60.31 ± 0.02	26.66 ± 0.02	28.88 ± 0.01	0.48 ± 0.02
15 (−1)	3.5 (0)	40 (−1)	87.8 ± 0.8	87.1 ± 0.4	38 ± 1	60.5 ± 0.6	1.20 ± 0.03	1.69 ± 0.02	18.4 ± 0.1	2.61 ± 0.04	59.17 ± 0.03	29.965 ± 0.007	36 ± 0	0.461 ± 0.008
45 (1)	3.5 (0)	40 (−1)	87 ± 3	90.6 ± 0.2	39.5 ± 0.7	60.9 ± 0.6	1.28 ± 0.03	1.83 ± 0.02	17.4 ± 0.3	2.68 ± 0.04	60.22 ± 0.07	29.67 ± 0.06	34.475 ± 0.007	0.47 ± 0.03
15 (−1)	3.5 (0)	80 (1)	47.31 ± 0.06	47.1 ± 0.3	37 ± 2	55 ± 2	1.40 ± 0.03	1.81 ± 0.01	21 ± 1	2.61 ± 0.04	62.43 ± 0.06	27.73 ± 0.06	35.17 ± 0.03	0.475 ± 0.009
45 (1)	3.5 (0)	80 (1)	38 ± 1	34.0 ± 0.5	35 ± 1	50.51 ± 0.05	1.27 ± 0.04	1.72 ± 0.02	24 ± 1	2.69 ± 0.04	57.81 ± 0.09	30.955 ± 0.007	37.63 ± 0.05	0.368 ± 0.008
30 (0)	2 (−1)	40 (−1)	89 ± 2	89.8 ± 0.6	39.1 ± 0.9	66.3 ± 0.8	1.13 ± 0.02	1.59 ± 0.01	13 ± 1	2.46 ± 0.06	57.5 ± 0.1	30.97 ± 0.03	44.45 ± 0.05	0.442 ± 0.003
30 (0)	5 (1)	40 (−1)	45.7 ± 0.7	45.3 ± 0.4	37.3 ± 0.5	55.8 ± 0.4	1.02 ± 0.03	1.51 ± 0.04	21.7 ± 0.1	2.93 ± 0.05	56.79 ± 0.08	29.88 ± 0.03	30.72 ± 0.01	0.47 ± 0.02
30 (0)	2 (−1)	80 (1)	35 ± 1	31.9 ± 0.5	31.33 ± 0.02	47 ± 1	1.09 ± 0.05	1.595 ± 0.008	17.9 ± 0.2	2.57 ± 0.04	53.655 ± 0.007	32.01 ±0.03	43.79 ± 0.03	0.41 ± 0.02
30 (0)	5 (1)	80 (1)	50 ± 1	50.4 ± 0.1	38.9 ± 0.4	56.7 ± 0.2	1.19 ± 0.04	1.73 ± 0.03	21.77 ± 0.07	2.48 ± 0.04	59.6 ± 0.0	28.85 ± 0.02	31.78 0.03	0.44 ± 0.01
30 (0)	3.5 (0)	60 (0)	44 ± 2	42.3 ± 0.9	37.14 ± 0.04	56.00 ± 0.07	1.16 ± 0.04	1.67 ± 0.01	22 ± 1.4	2.84 ± 0.05	61.6 ± 0.1	27.51 ± 0.03	34.54 ± 0.05	0.42 ± 0.01
30 (0)	3.5 (0)	60 (0)	45 ± 1	43.8 ± 0.4	38 ± 1	55.2 ± 0.4	1.19 ± 0.02	1.71 ± 0.02	21 ± 1	2.763 ± 0.007	56.825 ± 0.007	30.7 ± 0.0	35.03 ± 0.03	0.43 ± 0.01
30 (0)	3.5 (0)	60 (0)	39.08 ± 0.51	36.3 ± 0.1	36.4 ± 0.7	57.8 ± 0.6	1.13 ± 0.02	1.70 ± 0.02	17.7 ± 0.2	2.773 ± 0.007	55.24 ± 0.03	32.29 ± 0.02	35.62 ± 0.06	0.42 ± 0.02

Responses are informed as the mean and standard deviation ($n = 3$).

WRC: Water retention capacity, RW: Percentage of water retained, WHC: Water-holding capacity, SC: Swelling capacity, OHC: Oil holding capacity, WSF: Water soluble fraction.

TABLE 4. BOX-BEHNKEN DESIGN: COEFFICIENTS OF REGRESSION AND ANOVA FOR PROPERTIES OF DFC FROM PAPAYA PULP

	WHC (g/g)	SC (mL/g)	WRC (g/g)	RW (g/100g)	OHC (g/g)	Specific volume (mL/g)	WSF (g/100g)	*L**	*a**	*b**	Total polyphenols (g/100g)
Independent term	349.836	362.603	51.5122	104.089	1,62194	2.20027	−15.6334	69.9811	26.9731	73.6193	0,436563
A: Extraction time	−1.2052	−0.704462	0.142909	0.591542	−0,0297402	−0.0200439	0.0836197	−0.310111	0.281167	−0.187139	0,000667784**
B:Ethanol sample ratio	−19.2758	−20.4785	−2.11247	−5.27856	0,000308495	−0.01123429	10.5539	0.295556	2.17653	−10.8394**	−0,0377664**
C: Drying temperature	−7.53649**	−8.16464**	−0.399032*	−1.37266*	−0,00206179	−0.00986313	0.369733	−0.295042	−0.121927	−0.305219	0,00216863**
AA	0.0218994	0.0201479	−0.000913566	−0.00864907	0,000523586*	0.000424373	0.00271349	0.00535833	−0.003975	0.000867593	0,0000859976**
AB	0.114662	0.140832	0.0395107	0.0578889	0,00268987	0.00240821	−0.132611	0.0807222	−0.0573333	−0.027	−0,000217851*
AC	−0.00687169	−0.0138533	−0.00310614	−0.00389583	−0,000182084	−0.000194056	0.00330032	−0.00472083	0.00293333	0.00336667	−0,0000948842**
BB	−2.25859	−2.5845	−0.463199	−0.949907	−0,0286281	−0.0263473	−0.372181	−0.803056	−0.0202778	0.911759*	0,00794069**
BC	0.483097**	0.525616*	0.0782634*	0.166125*	0,00181899	0.00180142	−0.0383442	0.0557083	−0.0172083	0.014375	−0,0000281692
CC	0.0431927**	0.048387*	0.00119262	0.00577865	0,0000266852	0.0000907265	−0.00208748	0.00200781	0.000735937	0.0014224	−0,00000148054
R^2	91.91	91.87	81.10	92.94	95.11	79.28	85.57	62.19	63.87	98.81	84.18
Lack of fit (p)	0.0610	0.0923	0.1568	0.2007	0.4873	0.4592	0.6885	0.7774	0.9512	0.1886	0.0056

Significance levels (α): *0.05; **0.01.

WRC: Water retention capacity, RW: Percentage of water retained, WHC: Water-holding capacity, SC: Swelling capacity, OHC: Oil holding capacity, WSF: Water soluble fraction.

interaction between *E/S* and *Td*. These trends can also be observed in Fig. 1 (panels c, d), in which it is shown that an important increase in these properties occurred when *E/S* was low and the *Td* decreased, whereas at high *E/S* this trend reversed. Garau *et al.* (2007) found that the increase of the temperature of drying (hot air) promoted a clear decrease in the WRC values in DF extracted from peel and pulp of oranges and attributed this trend to the thermal degradation of highly hydrophilic polysaccharides present in DF (Kivelä 2011), and also with collapse and shrinkage phenomena.

The ability to retain oils has been related to the surface properties, to the overall charge density, to the structure and to the hydrophilic nature of the constituents (Elleuch *et al.* 2011). In the present research, only the quadratic coefficient of *t* had a positive and significant effect on OHC (Table 4).

Conversely, specific volume and WSF showed nonsignificant effects of the factors considered. These properties are of importance for the fiber characterization in terms of its functionality and nutritional quality. WSF may include water soluble compounds of low molecular weight, while specific volume may be relevant in relation to the bulking effects of fiber in the large intestine (Guillon *et al.* 2011). In particular, in the present research, OHC presented a positive linear correlation (index: 0.8832) with the specific volume. The same trend was observed by de Escalada Pla *et al.* (2010) for DF obtained from quince wastes and it had been attributed to the influence of structural characteristics on oil absorption.

The color parameter b^* represents the range of colors ranging from blue (negative b^* values) to yellow (positive b^* values). In Table 4, a significant and negative effect of *E/S* on b^* is observed which means that a higher *E/S* causes a loss of color, probably due to a loss of natural pigments such as carotenoids solubilized by the ethanol. Gayosso-García Sancho *et al.* (2011) identified various bioactive compounds including β-carotene, lycopene and β-criptoxanthin in fresh samples of pulp and peel of *C. papaya* L var. Maradol. Moreover, Craft and Soares (1992) reported solubility values in ethanol of 30 mg/L for β-carotene.

Finally, it can be stated that the total polyphenol content was strongly affected by the three factors considered (Table 4). The effect of *E/S* was negative and the *Td* and *t* effects were positive. Probably, an increase in *E/S* determined an increase in loss due to polyphenol solubilization (Spigno *et al.* 2007). The positive effect of *Td* may be due to substantial reductions in drying times because of the increase in temperature which can also contribute to the inactivation of residual enzymatic activity, for example of polyphenoloxidase. This positive effect of drying temperature on the content of phenolic compounds was also documented by Madrau *et al.* (2009) in apricots dried with hot air at different temperatures.

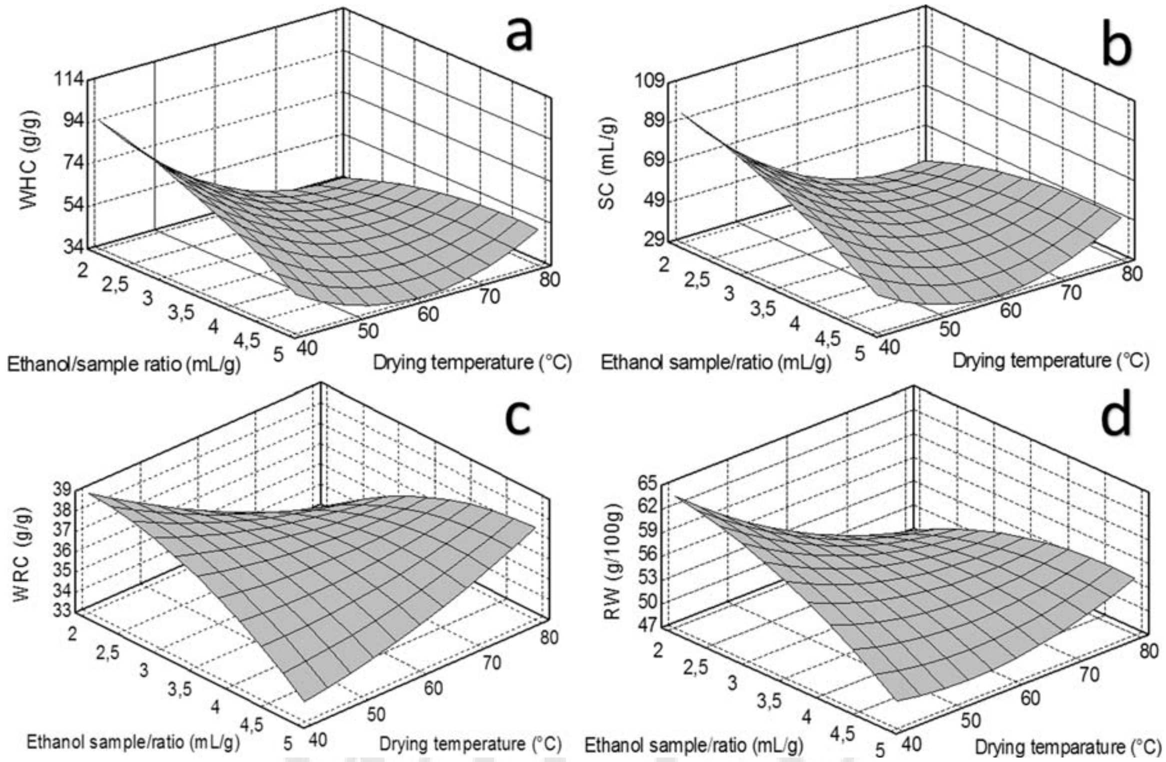

FIG. 1. RESPONSE SURFACES REPRESENTING THE EFFECT OF THE ETHANOL/SAMPLE RATIO AND THE MICROWAVE DRYING TEMPERATURE FOR A FIXED EXTRACTION TIME OF 15 MIN: (a) WATER HOLDING CAPACITY, WHC; (b) SWELLING CAPACITY, SC; (c) WATER RETENTION CAPACITY, WRC; (d) RETAINED WATER, RW

Optimization

The optimal conditions were studied with the aim of maximization of each response. It can be observed in Table 5, that the hydration properties (WHC, SC, WRC and RW) might be maximized by similar treatments, specifically applying higher *t*, low *E/S* and low *Td*. For other properties, the optimal process conditions were different. These results are important because they show the individual process conditions that might be needed to optimize each property along with the results estimated for each of them.

Multiple response analysis was performed to find the conditions of extraction and drying that optimize simultaneously several of the properties studied. This analysis included only WHC, SC, WRC, RW, OHC, specific volume, WSF, b^* and content of phenolic compounds, because these properties showed the best values for R^2. An approximate response value for each property was also statistically estimated for optimum conditions of treatment (Table 6). The multiple response analysis suggested an extraction time of 15 min with an *E/S* of 2.9 mL/g followed by a microwave drying at 40C. The desirability function for this method of extraction and drying was $d = 0.82$. The scale of the desirability function ranges between $d = 0$, for a completely undesirable response, and $d = 1$, for a fully desired response (Bezerra *et al*. 2008).

A new production process was performed for papaya pulp DFC with the conditions estimated by the multiple

TABLE 5. RESPONSE SURFACE METHODOLOGY: OPTIMIZATION OF RESPONSES

Property	Extraction time	Ethanol sample ratio	Drying temperature	Optimum value
WHC (g/g)	44.99	2.00	40.00	96.71
SC (mL/g)	45.00	2.03	40.00	100.91
WRC (g/g)	45.00	3.02	40.00	40.67
RW (g/100g)	31.87	2.00	40.00	66.15
OHC (g/g)	15.00	3.33	80.00	1.38
Specific volume (mL/g)	45.00	3.19	40.00	1.83
WSF (g/100g)	15.00	5.00	54.57	25.95
b^*	15.00	2.00	40.00	45.38
Total polyphenols (g/100g)	15.00	4.99	80.00	0.50

WRC: Water retention capacity, RW: Percentage of water retained, WHC: Water-holding capacity, SC: Swelling capacity, OHC: Oil holding capacity, WSF: Water soluble fraction.

TABLE 6. MULTIPLE RESPONSE ANALYSIS: PULP DFC PROPERTIES IN OPTIMAL CONDITIONS. COMPARISON WITH EXPERIMENTAL RESULTS FOR PULP AND PEEL DFC

Property	Results estimated by multiple response surface analysis for DFC of pulp	Experimental results for DFC of pulp	Experimental results for DFC of peel
WHC (g/g)	86.39	90.7 ± 0.9	27 ± 1
SC (mL/g)	85.11	84.0 ± 0.9	20.3 ± 0.2
WRC (g/g)	38.31	30.4 ± 0.4	21 ± 2
RW (g/100g)	61.52	46.3 0.7	39 ± 2
OHC (g/g)	1.23	1.20 ± 0.01	1.37 ± 0.03
Specific volume (mL/g)	1.68	1.64 ± 0.01	1.921 ± 0.006
WSF (g/100g)	16.90	24 ± 2	15 ± 1
b^*	39.84	37.61 ± 0.04	38.78 ± 0.03
Total polyphenols (g/100g)	0.44	0.47 ± 0.03	0.99 ± 0.03

WRC: Water retention capacity, RW: Percentage of water retained, WHC: Water-holding capacity, SC: Swelling capacity, OHC: Oil holding capacity, WSF: Water soluble fraction.

response analysis and the value of the product properties are presented in Table 6. In general, it can be seen, that the experimental results agreed with the statistical estimation for each property. This comparative analysis validated the statistical model used showing that prediction was adequate.

Papaya Peel DFC Production and Comparison with Pulp DFC Properties

DFC from peel of papaya was also produced with the conditions of the process above described. Experimental results for both DFCs are presented in Table 6.

Additionally, the AIR was evaluated for both optimized fractions. The AIR content of pulp and peel DFCs were 76.1 ± 0.6 and 83.1 ± 0.7 g/100g, respectively, showing that both fractions were enriched in cell wall polymers (Latorre *et al.* 2013).

The yield of peel DFC was 8.88 g/100g and it was greater than the one obtained for pulp DFC (2.56 g/100g). de Escalada Pla *et al.* (2012) reported DF yields of 2.6 and 4.6 g/100g for fractions obtained from peach (Calred variety) pulp and peel. Garau *et al.* (2007) also found a higher yield for DF obtained from orange peel than from pulp.

Higher ratios between WHC and WRC observed in the case of pulp can be attributed to its higher hydrophilicity originated in its greater galacturonic acid content which was 16 ± 1 g/100g while for peel DFC it was 11 ± 1 g/100g. WHC evaluates the water slightly associated with fiber matrix, which can have a beneficial effect on the body by increasing rapidly the stool weight (Cadden 1987) while WRC represents the water strongly retained after being subjected to external forces. DFC from pulp showed values of WHC and SC of 90.7 g/mL and 84.0 g/g, which are higher than those reported by Nieto Calvache *et al.* (2015) for DF isolated from peach bagasse. These results denote a very important water absorption and swelling capacity for the papaya pulp DFC, which allowed to conclude that this fiber can be used as a functional ingredient to modify the viscosity of aqueous systems, for example acting as a thickener of foods.

In the present research, it was also found that DFC of pulp and peel contained 24 and 15 g/100g, respectively, of WSF. Nieto Calvache *et al.* (2015) reported values of WSF between 11 and 16 g/100g for fractions enriched in peach DF obtained through different techniques. It is important to remark that a higher value of WSF might produce a lower T_g if WSF is composed mainly by small sugars.

In contrast, OHC and specific volume of pulp DFC, were lower ($P< 0.05$) than those of peel DFC (Table 6). The difference of OHC between both DFCs can be associated to the difference of specific volume, taking into account the positive Pearson correlation informed above. The results obtained for OHC ranged between were 1.20 g/g and 1.37 g/g for pulp DFC and peel DFC and are comparable with values ranging from 1.22 to 1.67 g/g reported by Gómez-Ordóñez *et al.* (2010), for DF isolated from several edible seaweeds from the northwestern Spanish coast.

The color is one of the sensory characteristics that must be considered when evaluating the adequacy of a new ingredient for the food industry (Tosh and Yada 2010). According to the results obtained (Tables 1 and 3), the drying temperature and the conditions of ethanol treatment produced significant changes in color. These changes might be related to certain reactions, such as enzymatic browning, nonenzymatic browning and caramelization (Krokida and Maroulis 2007).

The content of phenolic compounds determined as total polyphenols was 0.47 and 0.99 g/100g in DFC of pulp and peel, respectively (Table 6). Rivera-Pastrana *et al.* (2010) also found a greater content of phenolic compounds in exocarp of papaya (Maradol variety) than in mesocarp. Conversely, Saura-Calixto *et al.* (2007) and Hervert-Hernández *et al.* (2011) have reported values of 0.538 and 0.742 g/100g dry sample, as typical values for extractable polyphenols in the fruits of Spanish and Mexican diet.

The evaluation of the glass transition temperature, T_g, showed that pulp DFC and peel DFC had very different values for onset, midpoint and endpoint T_g, being the values for peel DFC, 28.03, 38.34, 43.75C and for pulp DFC, 5.52, 7.64, 9.15C. For papaya peel DFC, it was detected a midpoint T_g of $\sim$38C while for pulp DFC, the midpoint T_g was $\sim$8C and this difference is probably associated to the higher moisture content of pulp, 8.7 g/100g versus 6.6 g/100g for peel DFC. Also, pulp DFC showed a WSF value of 24 g/

100g, higher than the value of 15 g/100g observed for peel DFC trend that could have contributed to the lower T_g of the former if it is considered that the majority of the solids in WSF are simple sugars which are known to be responsible for the glass transition observed in fruits (Slade and Levine 1995; Vega-Gálvez *et al*. 2012). The high T_g observed for peel DFC along with its high water absorption capacity and cell wall polysaccharide content, indicates that this fraction can be used for improving the nutritional quality, rheological characteristics and thermochemical properties of food products. In particular, it can contribute to the shelf-life along storage of baked products such as cookies, helping to keep stable its organoleptic characteristics, which largely depend on the maintenance of their glassy state (Slade and Levine 1995), or also to increase the T_g in formulations of ice cream in which the crystallization phenomena are common during storage and distribution, impairing ice quality (Soukoulis *et al*. 2009).

CONCLUSIONS

The process conditions for the production of papaya pulp dietary fiber concentrates (DFCs), by means of ethanol extraction and microwave assisted drying, were studied through the use of a factorial design and a response surface analysis. The factorial design showed that the extraction parameters that exerted the greatest influence on DFC properties were the extraction time and ethanol/sample ratio.

The response surface analysis was performed using as process variables, the extraction time, ethanol/sample ratio and drying temperature. This study revealed that the process conditions that maximize hydration properties were similar (high time of extraction, low ratio of ethanol to sample and low drying temperature) while for the other properties evaluated, optimum conditions were different. The multiple response analysis proposed a technique for production of pulp DFC with optimized properties which involves a step of extraction of 15 min, with an ethanol/sample ratio of 2.9 mL/g and a drying temperature of 40C. Additionally, the same conditions were used to obtain DFC from papaya peel. Both DFCs obtained showed a high cell wall polymer content. It can be highlighted that the high water-holding and swelling capacity of the pulp DFC suggests the potential of this fiber for improving the rheological properties of aqueous systems. The glass transition temperature analysis showed higher T_g values for peel DFC suggesting its usefulness for improving thermochemical stability of foods products, helping to maintain the glassy state in processed foods. Finally, the content of phenolic compounds found in the DFC of peel was twice that found in the DFC of pulp, allowing to conclude that these concentrates may well provide some type of antioxidant activity when included in a food formulation.

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